Standard Operating Procedure for the Sampling and Analysis of Total Suspended Solids in Lake Water

LG302

Revision 04, December 2002

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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the sampling and analysis of Great Lakes Waters for total suspended solids (TSS). Samples of lake water are collected and filtered through a 0.7-µm pore-size glass fiber filter. Total suspended solids are operationally defined as the mass retained on the filter per unit volume of water.

2.0 SAFETY AND WASTE HANDLING

2.1 Refer to GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) and individual instrument procedural operations manuals for specific details on applicable 1) personal health and safety issues; 2) instrumental, chemical, and waste handling procedures; and 3) accident prevention. This applies to all EPA personnel, EPA contractors or federal, state, or local government agencies, and persons who operate or are passengers onboard US EPA GLNPO vessels during all activities and surveys.

2.2 It is the responsibility of the user of this method to comply with relevant chemical disposal and waste regulations as sited in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended). All applicable safety and waste handling rules are to be followed. All containers storing reagents, standards, controls, blanks, and wastes used in the laboratory must be properly identified through appropriate labeling and hazard definition. Good technique includes minimizing contaminated waste. Over-board discharges of chemical wastes are forbidden.

2.3 Every chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Please refer to Appendix L in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) for more detailed descriptions of the potential risks associated with any chemicals used in this method. It is good laboratory practice to wear a lab coat, safety goggles and gloves at all times.

2.4 During sampling, caution, common sense and good judgement should dictate appropriate safety gear to be worn in any given situation on deck. Hardhats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet; however, if in doubt, please ask the Chemical Hygiene Officer.

2.5 Collecting samples in cold weather, especially around cold water bodies, carries the risk of hypothermia and frostbite. Sampling team members should wear adequate clothing for protection in cold weather. For specific information regarding sampling during cold conditions, please refer to the US EPA GLNPO *Standard Operating Procedures for Winter Operations* (December 1994, or as amended).

2.6 Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

2.7 Work vests must be worn while working on the fantail and Rosette deck.

3.0 SUMMARY OF PROCEDURE

3.1 Great Lakes water samples are collected at pre-determined sampling stations and depths via either Rosette sampler or a submersible pump. Sub-samples of water are then filtered under vacuum through a 47-mm diameter glass fiber filter, which have been washed and dried to constant weight. The suspended solids are retained on the filter and frozen at -10°C until final weighing on an analytical balance in a land-based laboratory.

4.0 DESCRIPTION OF APPARATUS

4.1 Glass fiber filters are pre-weighed on an analytical balance. Water samples (typically 2 - 4 liters for open-lake locations) are collected from Rosette sampler, or an over-board pump. The filters are supported on a commercially-available, all-glass, 350-mL vacuum filtration apparatus. Final weights of the filters are determined identically to the initial weights. The equipment needed are listed in Table 1.

5.0 PREPARATION OF FILTERS AND ANALYTICAL BALANCE

5.1 Preparation of Filters

5.1.1 Filter preparation should take place as close to the start of the survey as possible.

5.1.2 Filters are to be handled only with stainless steel forceps. Filters that are mishandled after preparation should be discarded.

5.1.3 Label the 47-mm diameter GF/F filters (0.7-µm pore-size) using a permanent marker on the outer edge of each filter. Label from 1 - "X" with "X" being the total number of filters prepared. Allow the ink to dry for 5 minutes before proceeding to step 5.1.4.

5.1.4 Condition the filters using a 350 mL vacuum filtration apparatus. Pass 350 mL of reagent water through each filter. Place the filters onto the filtration apparatus with the labeled side facing up.

5.1.5 Remove the filters from the filtration apparatus and place them into individual 50-mm aluminum weighing pans. Dry the filters in a 105°C oven for 2 hours.

5.1.6 Remove the filters from the oven and place them into a desiccator. Allow the filters to cool for 5 minutes.

5.1.7 Prepare the analytical balance as described in section 5.2.

5.1.8 Remove the filters from the desiccator in small groups and weigh them on the analytical balance.

5.1.9 Record the initial filter weights on the TSS Sampling Log Sheet and place them individually into identically-numbered 50 mm diameter plastic petri dishes.

5.1.10 Every tenth filter must be re-dried in the 105°C oven for two hours and re-weighed. Record the second value on the log sheet.

5.1.11 If the second weight does not fall within ± 0.1 mg of the initial weight, check if the balance is zeroed correctly. If the balance has deviated, re-zero and re-weigh the filter. If the weight still does not fall within ± 0.1 mg, the previous group of 10 filters must be re-dried and re-weighed.

5.2 Preparation of Analytical Balance

5.2.1 A top-loading analytical balance with a capacity of 200 mg and a resolution of 0.01 mg is needed. The balance should be accompanied with a set of calibration weights, preferably NIST traceable (e.g., Mettler 22 balance with BA monitor).

5.2.2 Allow sufficient time for the analytical balance to warm up to operating temperature. Then, zero the balance.

5.2.3 Calibrate the balance using the 200 mg weight.

5.2.4 During the filter preparation and analysis procedures, the balance is zeroed after every tenth filter. If the balance deviates more than ± 0.05 mg, the balance is again zeroed, and the previous group of 10 filters are re-weighed.

6.0 FILTRATION AND ANALYSIS PROCEDURES

6.1 Filtration Procedure

6.1.1 Using stainless steel forceps, place one 47-mm GF/F filter onto the fritted glass support of the sampling apparatus. Place the glass funnel on top of the filter and secure with the clamp. Label the Great Lake name, station number, sampling depth, and date onto the petri dish.

6.1.2 Measure the volume of lake water to be filtered in a graduated cylinder, or mark four 1-liter Teflon bottles at the 1-liter level. Prior to filling, rinse the bottles or cylinder, twice with approximately 100 mL of lake water.

6.1.3 Connect the vacuum pump to the filtration flask. Maintain the vacuum between 5 - 10 inches of Hg during filtration. Add sample water to the filter funnel until enough suspended sediment has been collected (four liters or 20 minutes whichever comes first). Take care that the sample container is agitated before the addition of each increment so that a representative sample is filtered.

6.1.4 Record the volume of sample filtered.

6.1.5 After the lake water has been filtered, rinse the sides of the funnel with approximately 20 mL of reagent water and filter this rinse.

6.1.6 Remove the funnel, turn off the vacuum pump and relieve the vacuum. Using stainless steel forceps, place the filter back into the numbered petri dish. Place groups of petri dishes in a labeled Ziploc bag, and store at -10°C. Record the Great Lake name, station number, sampling depth, volume filtered, analyst, date, and time of day on the TSS Sampling Log Sheet.

6.1.7 Empty the filtrate from the filtration flask.

6.1.8 Rinse the filtration funnel, fritted glass support, and flask, and the container(s) with reagent water.

6.1.9 Re-assemble the filtration apparatus.

6.1.10 Place aluminum foil covers over the filtration funnel.

6.2 Analysis Procedure

6.2.1 Remove the filters from the freezer and allow them to thaw. Using stainless steel forceps, remove the filters from the petri dishes and place each in an individual 50-mm aluminum weighing pan. When handling the filters, grasp only the outer edges with the forceps.

6.2.2 Dry the filters in a 105°C oven for two hours.

6.2.3 Prepare the analytical balance (section 5.2).

6.2.4 Using the same analytical balance as the initial weighing procedure, follow steps 5.1.6 - 5.1.12 to determine the final weights of the filters.

6.2.5 Store the filters in a freezer after all of them have been weighed and the results recorded.

6.2.6 Calculate the total suspended solids (TSS) as:

$$\textit{Total Suspended Solids } (mg/L) = \frac{\textit{Final weight} - \textit{Initial weight}}{\textit{Sample volume in liters}}$$

7.0 QUALITY CONTROL

7.1 Refer to the Chapter 3 Introduction for definitions of quality control samples and information regarding quality control procedures, such as QC sample IDs and labeling.

7.2 The following QC samples are prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria
High Check Standard (CH)	Not required but being considered	Not applicable
Low Check Standard (CL)	Not required but being considered	Not applicable
Field Reagent Blank (FRB)	At least once during the sampling of each Great Lake	± 0.2 mg/L
Lab Duplicate (LD1)	At least once during the sampling of each Great Lake	± (0.2 mg/L + 0.2*mean)

7.2.1 A duplicate sample will be filtered in parallel at least once during the sampling of each Great Lake.

7.2.2 A TSS field blank will be collected at least once during the sampling of each Great Lake. A TSS field blank is collecting by filtering 1 L of blank water. The filter after filtration processed in the same manner as a sample.

7.3 Assessment

7.3.1 The analyst must compare analytical results to the acceptance criteria listed in Section 7.2 to identify QC failures. If the results are outside the acceptance criteria, the analyst should first review their calculations for errors and if none are identified, they must follow the corrective action procedures listed in Section 7.4.

7.4 Corrective Actions

7.4.1 Corrective action procedures, because of the nature of the analytical procedure, are limited to verifying procedures and calculations prior to flagging the associated results and reporting the deviations to GLNPO QA officer. The analyst should check transcriptions, calculations, instrument calibrations, instrument sensitivities, and any other potential sources of error. If failure occurs and no error is identified, the analyst must enter the appropriate remark code in the data associated with the failed QC unit and report the deviation to the GLNPO project officer or QA officer.

8.0 REFERENCES

8.1 Standard Operating Procedure for the sampling and analysis of total suspended solids in Great Lakes waters. GLNPO Organics SOP-04, 06/01/94: Rev. 1.

Table 1: List of Filtration Equipment

Quantity	Equipment
2	Teflon wash bottles
2	350-mL all-glass filtration apparatus
2	Stainless steel forceps
2	Support/ring stand for filtration apparatus
1	Top-loading analytical balance 200 mg capacity, 0.01 mg resolution
1	Calibration weights
1	Desiccator
1	Drying oven

Miscellaneous (some quantities depend on number of samples)
47-mm GF/F filters (0.7 µm pore-size) Whatman 1825-47
Cubitainers
Tygon tubing (3/8"ID)
50-mm diameter aluminum weighing pans
50-mm diameter plastic Petri dishes
Permanent markers
Ziploc freezer bags